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(54) Title: <b>NON-MELTING GELLAN GUM GEL PIECES CONTAINING MILK SOLIDS</b>			
(57) Abstract  The invention is a heat-stable gel composition comprising about 0.1-1 % gellan gum, about 1.0-10 % milk solids, about 0.05-0.2 % calcium salt, and water. Preferably, the composition comprises about 0.15-0.5 % gellan gum, about 2.3-5 % milk solids, about 0.1-0.15 % calcium salt, and water. Optionally, compositions of the invention include about 0.01-0.1 % sequestrant.			

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**TITLE OF THE INVENTION****NON-MELTING GELLAN GUM GEL PIECES CONTAINING MILK SOLIDS****5     BACKGROUND OF THE INVENTION**

It is well known that gellan gum requires ions for gelation. Depending on ion type and ion concentration, such gels can be made which either re-melt or do not re-melt on heating.

10     Gellan gum gels formed in gel pieces can be used as components in beverages and other aqueous environments. Provided the gel pieces are allowed to remain in the ion forms that are non-melting, the pieces, when present in these aqueous environments, do not re-melt on subsequent heat treatment.

15     Japanese Publication 1037258 describes gels, containing gellan gum and sodium chloride salt, calcium lactate salt, magnesium chloride salt, sodium phosphate salt, and/or organic acids including citric acid or malic acid, which are heat resistant. The publication does not describe heat stable gel pieces immersed in a liquid and does not describe heat stable gel pieces containing milk solids.

20     Japanese Publication 1039955 describes gels, containing gellan gum and optionally other gums, including xanthan gum, carrageenan or guar gum along with sodium chloride, calcium lactate, magnesium chloride, and/or potassium chloride, which are heat resistant. The publication does not describe heat stable gel pieces immersed in a liquid and does not describe heat stable gel pieces containing milk solids.

25     Japanese Publication 1060338 states that gellan, optionally along with starch, agar, alginic acid, dextran, and guar gum, provides increased heat resistance for xanthan gum and locust bean gum. The publication does not describe heat stable gel pieces immersed in a liquid and does not describe heat stable gel pieces containing milk solids.

30     While gellan gum gel pieces formed with preselected ion types and concentrations have been prepared and are thermostable, gel pieces containing milk solids are difficult to form and generally not heat stable. Following procedures described in the prior art, where the source

of calcium is added at or near the boiling point of the gellan gum solution, milk protein precipitates prior to formation of the gel. The resulting gel is a non-homogeneous melting gel which breaks down following exposure to retort conditions (120°C for 20 minutes). Retort conditions are known in the art as those conditions used to sterilize foods.

The present invention provides new gellan gum gel pieces containing milk solids which are retort-stable, and solves the stability problem which arises when it is attempted to incorporate milk solids, including milk protein, in a gellan gum gel piece.

### SUMMARY OF THE INVENTION

The invention is a heat-stable gel composition comprising about 0.1 - 1% gellan gum, about 1.0 - 10 % milk solids, about 0.05 - 0.2% calcium salt, and water. Preferably, the composition comprises about 0.15 - 0.5% gellan gum, about 2.3 - 5% milk solids, about 0.1 - 0.15% calcium salt, and water. Optionally, compositions of the invention include about 0.01 - 0.1% sequestrant.

The invention is also a heat stable gel piece comprising about 0.1 - 1% gellan gum, about 1.0 - 10 % milk solids, about 0.05 - 0.2% calcium salt, and water, wherein the piece has a volume of between about 0.5 and 2 cm<sup>3</sup>, and a weight of between about 0.5 and 2 grams. Preferably, the gel piece composition comprises about 0.15 - 0.5% gellan gum, about 2.3 - 5% milk solids, about 0.1 - 0.15% calcium salt, and water.

### DETAILED DESCRIPTION OF THE INVENTION

In general, non-melting gellan gum pieces are prepared by hydrating gellan gum in an aqueous medium under appropriate process conditions, adding a source of calcium ions, allowing the mixture to cool to form a gel, and cutting the gel into pieces or cubes.

Gel pieces of the present invention can be used in products such as hot or cold acid or non-acid beverages, including coffee, tea, juices, colas, dairy drinks, and also in non-beverage products such as retorted snacks (canned foods containing soft cubes of protein gels, e.g.

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leche flan, almond milk), dessert toppings, and puddings. Retort stable protein gels, including gelatine, dairy, soymilk, coconut milk, and other protein systems, may also be produced using the gels pieces of the invention.

5                   Equilibration takes place between the solutes in the gel and the surrounding aqueous medium when the pieces are placed in the surrounding aqueous medium. Since gellan gum is an ion exchanger, such equilibration results in ion-exchange. Depending on how the pieces have been formulated and the composition of the surrounding aqueous  
10 medium, such ion exchange can convert the gel from a non-melting to a melting gel which melts upon heating. By providing the proper balance of ions in the gel pieces and the surrounding aqueous medium, the gel piece, after equilibration, remains in non-melting form.

15                   According to the present invention, heat-stable gellan gum gel pieces containing protein are prepared by adding the gelling ion source at a temperature well below the boiling point of the gellan gum-containing and protein-containing pre-gel solution. Incorporation of the calcium source in this way avoids protein precipitation and yet induces  
20 heat stable gel formation. The formed gel is then cut into pieces of desired shape and size.

25                   The gel pieces of the present invention are comprised of the following ingredients and amounts (amounts are weight percent unless otherwise indicated). The amount of water is that present in the formed gel matrix.

	<u>Ingredient</u>	<u>Amount</u>
	Gellan gum	about 0.1-1%
	Milk solids	about 1.0-10 %
	Calcium salt	about 0.05-0.2%
30	Water	Q.S.

Optionally, sequestrants such as sodium citrate, at levels of up to 0.1% (e.g. between 0.01 - 0.1%) are used to facilitate gel formation and stability.

Preferably, the gel pieces of the present invention are comprised of the following ingredients and amounts (amounts are weight percent unless otherwise indicated). The amount of water is that present in the formed gel matrix.

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<u>Ingredient</u>	<u>Amount</u>
Gellan gum	about 0.15-0.5%
Milk solids	about 2.3-5%
Calcium salt	about 0.1-0.15%
Water	Q.S.

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Optionally, sequestrants such as sodium citrate, at levels of up to 0.1% (e.g. between 0.01 - 0.1%) are used to facilitate gel formation and stability.

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Gellan gum is a heteropolysaccharide prepared by fermentation of *Pseudomonas elodea* ATCC 31461. Gellan gum is available from Kelco Division of Merck & Co., Inc., under various names, including KELCOGEL and KELCOGEL F. Processes for preparing gellan gum include those described in United States Patents 4,326,052 and 4,326,053. It is useful for a variety of gelling, texturing, stabilizing and film forming applications, particularly as a gelling agent in foods, personal care products and industrial applications.

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According to The Merck Index, 11th Edition, 1989, page 6118, the composition of cow's milk is about 3.82% fat, 3.25% protein, 4.64% lactose, 0.73% ash, and water. Liquid cow's milk contains about 12.43% solids. Therefore, the extracted solid component of milk comprises approximately 31% fat, 26% protein, 37% lactose, and 6% ash. The composition of extracted milk solids can be altered by various common processing techniques to reduce fat content (i.e. increase protein content). Partial removal of fat, from 31% to 15%, provides remaining milk solid components of 32% protein, 46% lactose, and 7% ash. Complete fat removal provides an extracted solid component comprising 38% protein, 54% lactose, and 8% ash. For purposes of the present invention, "milk solids" refers to a solid component of milk comprising

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between about 0-15% fat, 32-38% protein, 46-54% lactose, and 7-8% ash.

Examples of commonly available milk solids are skim milk powder, instant milk powder, reconstituted milk powder, and modified milk powder. Imitation milk powder (also referred to as milk substitutes or synthetic milks), although not directly derived from common milk sources, also falls within the meaning of the term "milk solids" for purposes of the present invention. Skim milk powder is essentially fat-free. Other powders may contain a fat component. Skim milk powder, instant milk powder, reconstituted milk powder, modified milk powder, and imitation milk powder are all described in Caric, "Concentrated and Dried Dairy Products" VCH Publishers, Inc., New York, NY, pp. 97-125, (1994).

Coffee whiteners are compositions containing milk solids, derived from condensed milk (which contain about 25% milk solids), added sugar and stabilizers. These components are combined and dried. They are usually prepared with condensed skim milk but may contain fat. Additional examples of commonly available milk solids for preparing coffee whiteners are condensed unsweetened condensed milk, sweetened condensed milk, unsweetened condensed skim milk, sweetened condensed skim milk, unsweetened flavored condensed milk, sweetened condensed skim milk, "block" milk, and caramelized condensed milk. These are all described in Caric, "Concentrated and Dried Dairy Products" VCH Publishers, Inc., New York, NY, pp. 7-55, (1994). "Coffeemate" is a coffee whitener which is commercially available from Carnation.

Calcium salts suitable for use in the gels of the present invention include calcium lactate, calcium chloride and calcium sulfate.

Typical sequestrants that can be employed in forming the gels of the present invention include a variety of inorganic phosphates such as sodium hexametaphosphate, tetrasodium pyro-phosphate, disodium orthophosphate, and sodium tripolyphosphate, sodium citrate and EDTA.

Typical acidulants, such as citric acid or malic acid, sweeteners, such as natural and artificial sweeteners, preservatives, such as sodium benzoate, flavorings such as flavor emulsion oil bases, including coffee, tea, grape, orange, apple, or lemon flavors, other  
5 common beverage additives such as ascorbic acid, and colorings, may also be used in the gels of the present invention.

The gels are generally prepared according to the following procedure:

- 10 1. add dry gellan gum to water with stirring and heat to between about 90 and 100°C until gellan gum is hydrated to form a solution;
2. cool the solution to between about 65 and 80°C and add milk  
15 solids;
3. dissolve calcium salt in water, and add the calcium salt solution to the gellan gum/ milk mixture at between about 60 and 70°C with stirring; and
- 20 4. deposit the composition formed in step 3 into a gelling vessel, and allow the composition to gel.

It is a critical feature of the process for preparing the non-melting protein-containing gel pieces of the present invention that the  
25 calcium salt be added at a temperature well below the boiling point of the gum solution. The addition of calcium at high temperatures results in formation of undesirable protein precipitates as well as a gel which is not heat stable.

Optionally, sequestrants such as disodium phosphate, sodium citrate and trisodium phosphate can be dry blended with gellan  
30 gum prior to addition of gellan gum to water. Sequestrants enhance the process by providing greater flexibility in determining appropriate temperature conditions during gel preparation. These sequestrants reduce protein precipitation at elevated temperatures.



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Also, sugar can optionally be dry blended with gellan gum prior to addition of gellan gum to water. Sugar enhances the dispersion of gellan gum during addition to water.

5 Following gel formation, the gel is cut into cubes by conventional means and added to the desired beverage, e.g. coffee, tea, etc., or food product.

Retort stability was measured by subjecting the formed gel pieces to 120°C. for 20 minutes and observing the resulting effect on the integrity of the gel pieces. Those gel pieces considered retort stable are  
10 those which retain their gel structure following exposure to these conditions.

### EXAMPLE 1 (CONTROL)

#### Thermostable gel

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The following gel piece was prepared with water, calcium lactate, gellan gum (KELCOGEL, commercially available from Kelco, a Division of Merck & Co., Inc., San Diego, CA), and sodium chloride. The gel piece does not contain protein or milk solids.

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<u>Ingredients</u>	<u>grams</u>	<u>% (weight)</u>
water (DI)	496.45	99.29
calcium lactate	1.55	0.31
KELCOGEL	1.50	0.30
25 sodium chloride	0.50	0.10

Gellan gum was dispersed in water and the dispersion was heated to 80 degrees C. After the gum hydrated, forming a solution, sodium chloride and calcium lactate were added. The solution was  
30 poured into a flat, shallow pan and allowed to cool. After the gel formed, the gel was cut into cubes.

The cubes were heat stable (they withstood pasteurization @ 80 degrees C. for 20 minutes) and did not lose their integrity in various liquids in which equilibration between the solutes in the gel pieces and

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surrounding medium did not result in conversion of the gel from a non-melting to melting form e.g. Kool-Aid™ (Kraft general Foods), Gatorade™ (The Quaker Oats Co.).

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### EXAMPLE 2 (CONTROL)

#### Thermostable gel

The following gel piece was prepared with water, calcium lactate, gellan gum, sodium chloride, xanthan gum (Keltrol T, commercially available from Kelco, a Division of Merck & Co., Inc., San Diego, CA), and clear locust bean gum (Gelloid LB 230, available from FMC). The gel piece does not contain protein or milk solids.

	<u>Ingredients</u>	<u>grams</u>	<u>% (weight)</u>
15	water (DI)	495.95	99.19
	calcium lactate	1.55	0.31
	KELCOGEL	1.50	0.30
	sodium chloride	0.50	0.10
	Keltrol T	0.25	0.05
20	Gelloid LB 230	0.25	0.05

Gellan gum, xanthan gum, and locust bean gum were dispersed in water and the dispersion was heated to 80 degrees C. After the gum hydrated, forming a solution, sodium chloride and calcium lactate were added. The solution was poured into a flat, shallow pan and allowed to cool. After the gel formed, the gel was cut into cubes.

The cubes were heat stable (they withstood pasteurization @ 80 degrees C. for 20 minutes) and did not lose their integrity in various liquids in which equilibration between the solutes in the gel pieces and surrounding medium did not result in conversion of the gel from a non-melting to melting form e.g. Kool-Aid™ (Kraft general Foods), Gatorade™ (The Quaker Oats Co.).

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**EXAMPLE 3 (CONTROL)****Thermostable gel**

5 This formulation produces excellent jelly cubes with outstanding transparency and good heat stability.

	<b><u>INGREDIENTS</u></b>	<b><u>GRAMS</u></b>	<b><u>PERCENT</u></b>
	Water	421.00	84.20
10	Sugar, granular	75.00	15.00
	KELCOGEL gellan gum	2.00	0.40
	Magnesium chloride ( $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ ) 10%	1.50	0.30
	Sodium chloride, 10%	<u>0.50</u>	<u>0.10</u>
15		500.00 g	100.00%

20 KELCOGEL was mixed with approximately 5 times its weight of sugar. With mechanical agitation, the KELCOGEL/sugar blend was dispersed in tap water. The mixture was heated to boiling and held at boiling for 1 minute to ensure gum hydration. Magnesium chloride and NaCl were added and mixed for 1 minute. The remainder of the sugar was gradually added and mixed for 3 minutes while maintaining heat to ensure the sugar was dissolved. Coloring can be added if needed and mixed at the same temperature for 1 minute. The solution was poured into rectangular molds and cooled at room temperature or under refrigeration. The product was cut into diced pieces.

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**EXAMPLE 4****Thermostable gelled coffee whitener**

The following composition was prepared:

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<u>Ingredient</u>	<u>weight %</u>
sugar	9.00
Coffeemate™	2.00
skim milk powder	1.00
KELCOGEL F gellan gum	0.35
calcium lactate	0.15
water	87.50

15

KELCOGEL F gellan gum and sugar were dry blended. The blend was added to boiling water with continuous stirring until the gellan gum was fully hydrated. The hydrated solution was cooled to 70°C. Skim milk powder and coffee whitener were added.

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Calcium lactate, dissolved in water, was then added to the solution at 65°C with continuous stirring. The resulting product was deposited into a suitable gelling vessel and left to form a gel.

After gel formation, the gel was cut into cubes. The cubes were subjected to 120°C for 20 minutes in coffee solution and found to be stable.

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**EXAMPLE 5****Thermostable gelled coffee whitener**

The following composition was prepared:

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	<u>Ingredient</u>	<u>weight %</u>
	sugar	9.000
	Coffeemate™	2.000
	skim milk powder	1.000
10	KELCOGEL F gellan gum	0.350
	calcium lactate	0.200
	sodium citrate	0.075
	water	87.375

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KELCOGEL F gellan gum, sodium citrate and sugar were dry blended. The blend was added to boiling water with continuous stirring until the gellan gum was fully hydrated. The hydrated solution was cooled to 70°C. Skim milk powder and coffee whitener were added.

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Calcium lactate, dissolved in water, was then added to the solution at 65°C with continuous stirring. The resulting product was deposited into a suitable gelling vessel and left to form a gel.

After gel formation, the gel was cut into cubes. The cubes were subjected to 120°C for 20 minutes in a coffee solution and found to be stable.

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**EXAMPLE 6**

Example 4 was repeated except that the level of calcium lactate was 0.1% and addition of calcium lactate was conducted at temperature near boiling. Protein precipitated prior to gel formation.

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The formed gel was not retort stable.

EXAMPLE 7

Example 6 was repeated except that the level of calcium lactate was 0.15% and addition of calcium lactate was conducted at temperature near boiling. Protein precipitated prior to gel formation. The formed gel was not retort stable.

EXAMPLE 8

Example 6 was repeated except that the level of calcium lactate was 0.2% and addition of calcium lactate was conducted at temperature near boiling. Protein precipitated prior to gel formation. The formed gel was not retort stable.

EXAMPLE 9

Example 5 was repeated except that 1) the level of calcium lactate was 0.2%, 2) the addition of calcium lactate was conducted at 80°C, and 3) the sequestrant used was disodium phosphate at a level of 0.1%. Some protein precipitation occurred prior to gel formation. The formed gel was not retort stable.

EXAMPLE 10

Example 9 was repeated except the sequestrant used was sodium citrate. Some protein precipitation occurred prior to gel formation. The formed gel was not retort stable.

EXAMPLE 11

Example 9 was repeated except the sequestrant used was trisodium phosphate. Some protein precipitation occurred prior to gel formation. The formed gel was not retort stable.

EXAMPLE 12

Example 4 was repeated except that the level of calcium lactate was 0.1%. Protein did not precipitate prior to gel formation. The formed gel was slightly unstable when exposed to retort conditions.

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**EXAMPLE 13****Thermostable gelled coffee whitener**

5        Example 12 was repeated except that the level of calcium lactate was 0.12%. Protein did not precipitate prior to gel formation. The formed gel was retort stable.

**EXAMPLE 14****Thermostable gelled coffee whitener**

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Example 12 was repeated except that the level of calcium lactate was 0.2%. Slight protein precipitation occurred prior to gel formation. The formed gel was retort stable.

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**EXAMPLE 15**

Example 5 was repeated to evaluate the effect of sequestrant amount and type on a system containing 0.2% calcium lactate. The results are summarized below:

20	<u>Sequestrant</u>	<u>weight %</u>	<u>Precipitate</u>	<u>Gel</u>	<u>Retort stable</u>
	sodium citrate	0.05	yes (fine)	yes	no
		0.075	no	yes	yes
		0.10	no	yes	no
25	disodium phosphate	0.05	yes	yes	no
		0.075	no	yes	yes
		0.10	no	yes	no
30	trisodium phosphate	0.05	yes (very fine)	yes	no
		0.075	no	yes	-
		0.10	no	no	-

**EXAMPLE 16 (CONTROL)****Thermostable gel**

5                   KELCOGEL Gellan gum, KELTROL xanthan gum and locust bean gum are combined and used to form nearly clear gel cubes for novel beverages. These cubes have excellent heat stability (withstand pasteurization @ 80°C for 20 minutes) in liquids such as juice drinks, without losing their integrity.

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<u>INGREDIENTS</u>	<u>GRAMS</u>	<u>PERCENT</u>
Tap water (up to 300 ppm hardness)	445.70	89.14
Sucrose	50.00	10.00
15   KELCOGEL gellan gum (Kelco)	1.50	0.30
KELTROL xanthan gum (Kelco)	0.25	0.05
locust bean gum	0.25	0.05
Calcium lactate pentahydrate (J.T. Baker)	1.55	0.31
Salt	0.50	0.10
20   Sodium citrate dihydrate powder	<u>0.25</u>	<u>0.05</u>
(Haarmann & Reimer)		
	500.00 g	100.00%

25                   Sugar, gums and sodium citrate were dry blended and added to water under efficient agitation. The mixture was heated to a rolling boil, salts added, and mixed for 15-30 seconds. Brix was adjusted to 10° (to compensate for evaporation), and the hot solution poured into flat shallow pan(s), or mini ice cube trays, and cooled. Gels were cut into cubes, and cubes added to the beverage. The beverage and cubes were  
30                   pasteurized at 80°C for 20 minutes.



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Sucrose may be varied to adjust the brix of the gel to equal that of the beverage into which the gel cubes will be placed. If very hard water is used (>300 ppm hardness, calculated as  $\text{CaCO}_3$ ), additional sodium citrate may be needed to assure complete hydration of the gums.

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WHAT IS CLAIMED IS:

1. A heat-stable gel composition comprising about 0.1 - 1% gellan gum, about 1.0 - 10 % milk solids, about 0.05 - 0.2% calcium salt, and water.
2. A composition of Claim 1 comprising about 0.15 - 0.5% gellan gum, about 2.3 - 5% milk solids, about 0.1 - 0.15% calcium salt, and water.
3. A composition of Claim 1 comprising about 0.1 - 1% gellan gum, about 1.0 - 10 % milk solids, about 0.05 - 0.2% calcium salt, 0.01 - 0.1% sequestrant, and water.
4. A composition of Claim 3 comprising about 0.15 - 0.5% gellan gum, about 2.3 - 5% milk solids, about 0.1 - 0.15% calcium salt, about 0.01 - 0.1% sequestrant., and water.
5. A heat stable gel piece comprising a gel composition of Claim 1, a volume of between about 0.5 and 2 cm<sup>3</sup>, and a weight of between about 0.5 and 2 grams.
6. An aqueous fluid beverage comprising water, flavorings, and gel pieces of Claim 1.
7. A food product comprising gel pieces of Claim 1.
8. A process for preparing a heat stable gel piece comprising about 0.1 - 1% gellan gum, about 1.0 - 10 % milk solids, about 0.05 - 0.2% calcium salt, and water, which process comprises
  - a) adding dry gellan gum to water with stirring and heat to between about 90 and 100°C until gellan gum is hydrated to form a solution;

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- b) cooling the solution to between about 65 and 80°C and adding milk solids;
- c) dissolving calcium salt in water, and adding the calcium salt solution to the gellan gum/ milk mixture at between about 60 and 70°C with stirring;
- d) depositing the composition formed in step (c) into a gelling vessel, and allowing the composition to gel; and
- e) cutting pieces from the gel formed in step (d).

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# INTERNATIONAL SEARCH REPORT

Intern. Application No

PCT/US 95/13782

A. CLASSIFICATION OF SUBJECT MATTER  
 IPC 6 A23L1/054 A23C11/00 A23L2/52

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 A23L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	GB,A,2 261 805 (KRAFT GENERAL FOODS) 2 June 1993 see page 6, line 4 - line 16 see page 3, paragraph 2 see claims 1,2 ---	1,2
A	B.H.WEBB: 'Fundamentals of Dairy Chemistry' 1965, AVI, WESTPORT - CONNECTICUT Page 17 see table 8 ---	
X	EP,A,0 607 002 (MERCK & CO.) 20 July 1994 see page 6; table ---	1,3
E	EP,A,0 685 171 (UNILEVER) 6 December 1995 see claims 1,5,7,8 ---	1,2
	-/-	

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Name and mailing address of the ISA

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# INTERNATIONAL SEARCH REPORT

Int. No. Application No  
/US 95/13782

## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>PATENT ABSTRACTS OF JAPAN vol. 011 no. 371 (C-462) ,3 December 1987 &amp; JP,A,62 143653 (SAN EI CHEM IND LTD) 26 June 1987, see abstract &amp; DATABASE WPI Week 8731 Derwent Publications Ltd., London, GB; AN 87-217594 &amp; JP,A,62 143 653 see abstract</p> <p>---</p>	
A	<p>PATENT ABSTRACTS OF JAPAN vol. 013 no. 211 (C-597) ,17 May 1989 &amp; JP,A,01 030547 (ASAI IWA0) 1 February 1989, see abstract &amp; DATABASE WPI Week 8911 Derwent Publications Ltd., London, GB; AN 89-079971 &amp; JP,A,01 030 547 see abstract</p> <p>-----</p>	

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Information on patent family members

Intern: I Application No

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